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The integrated process of microwave torrefaction and pyrolysis of corn stover for biofuel production

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ABSTRACT

The integrated process of torrefaction and pyrolysis of corn stover were investigated in this study. The effects of torrefaction conditions on both torrefaction and pyrolysis products were determined. The heating values of torrefied biomass were similar to those of coals and increased comparing to raw biomass. The torrefaction oils contained high value chemicals such as furans and phenolics. The torrefaction process caused a decrease in the pyrolytic oil yields but the total bio-oil yield from torrefaction and pyrolysis were similar to those from raw biomass pyrolysis. The pyrolytic oil from torrefied biomass contained more hydrocarbons and phenols and less organic acids than raw biomass pyrolysis, which indicates that the quality of pyrolytic oil was improved with torrefaction as a thermal pretreatment. The optimum torrefaction condition for both high yield and quality of pyrolytic oil was determined at the reaction temperature around 275 °C and torrefaction time of 30 min. This study implied that torrefaction as a pretreatment can be combined with the pyrolysis for high quality biofuel production.

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1. Introduction

Biomass is one of the most abundant and renewable energy sources. In today's world 12% energy is supplied by biomass resources in form of generating electricity and producing ethanol and high energy gas [1–4]. During the last decade the exploration of using biomass and biomass treatments is becoming a topic of greater interest. Among the biomass conversion technologies, pyrolysis is one promising technology to produce liquid fuel which has some similar properties with crude oil and potentially can be used to replace the current carbon fuel after upgrading [5,6]. However, biomass is complex and has high water content and low energy density. These properties lead to the low quality of bio-oil such as low heating value, high organic acid content, and large active chemical compounds. Bio-oils are unstable during storage due to their physical and chemical properties and are difficult to be used as transport fuels. The refinery approaches and upgrading methods have been explored to improve the quality of bio-oils, however additional costs are required for such post-pyrolysis upgrading [7–9].

Torrefaction is a mild thermal treatment method that is operated at a temperature ranging from 200 to 300 °C in the absence of oxygen [10]. In the torrefaction process water is removed and the biomass is thoroughly dried. The hemicelluloses are decomposed and the cellulose and lignin are dehydrated. The structure and composition of biomass are modified after the torrefaction. Previous reports pointed out that the O/C ratio of biomass is decreased and the energy content is increased during torrefaction [11,12] thereby improving the properties of torrefied biomass. Numbers of reports in biomass torrefaction pointed out that the torrefied biomass as solid fuel can be comparable with coals [13–17]. Several papers also pointed out that torrefaction as pretreatment in gasification and combustion improved the properties of syngas and the efficiency of electricity generation [11,12,18,19]. Recently a couple of papers reported that the torrefaction conditions have effects on the woody biomass pyrolysis behaviors and their products [16,17,20].

Microwave pyrolysis is one of the thermo-chemical technologies whereby biomass is heated by microwave irradiation. Due to fast internal and volume heating by microwave irradiation, microwave pyrolysis has been widely investigated using crop residue and woody biomass [4,16,17,21–24]. Recently microwave torrefaction for woody biomass and crop residues has been studied [16,17,25,26]. However, few results were reported in the integrated process of microwave torrefaction and pyrolysis in biomass.

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The objective of this study was to investigate the integrated process of microwave torrefaction and pyrolysis for corn stover. The effects of microwave torrefaction conditions on pyrolysis and products were determined. The chemical composition of bio-oil from both the torrefaction and pyrolysis was characterized by GC/MS. The mechanism of microwave torrefied biomass is discussed briefly.

2. Materials and methods

2.1. Materials

Corn stover used in this study was collected from Brookings, South Dakota, USA. Corn stover was ground with 4 mm screening by Thomas-Wiley lab mill (Model No. 3375-E15, Thomas Scientific, USA) before the process. Composition of each material, including structural carbohydrates, lignin, and moisture content, was measured according to NREL/TP-510-42618 procedure. The corn stover contained 7.94 ± 0.23% moisture content, 36.38 ± 0.44% glucose, 16.99 ± 0.19% xylose, and 16.29 ± 1.01% lignin.

2.2. Microwave torrefaction and pyrolysis

A MAS-Π batch microwave oven (Shanghai Microwave Chemistry Technology Co., Ltd., China) with a water condenser system was used for both corn stover torrefaction and pyrolysis in this study. Details of the set-up can be found in previous research [16,17,24].

Microwave torrefaction of corn stover was performed at a 400 W power setting. 100 g of corn stover loaded in a one liter quartz flask was placed inside of the oven. The N_2 flow passed through the system for 20 min prior to treatment to create an inert oxygen atmosphere. The volatiles were condensed by a water-condenser system with five half meter long parallel bulb condensers connected to the flask. The non-condensable gases were collected at the end of the condenser, and the torrefied biomass was left in the flask. The weight of torrefied biomass was measured after cooling to room temperature. The weight of non-condensable gases can be obtained based on the mass balance as shown in Eq. (1).

$$\begin{aligned}&\text{weight of torrefaction noncondensable gas}\\&\quad= 100\,\text{g} - \text{weight of torrefaction oil}\\&\quad- \text{weight of torrefied biomass}\end{aligned} \tag{1}$$

Torrefied biomass pyrolysis was performed at the reaction temperature of 650 °C for15 min with the power setting at 700 W based on previous studies [24]. After the reaction, the pyrolytic oil and biochar were collected and weighed. The pyrolysis noncondensable gas was calculated based on the mass balance shown in Eq. (2).

$$\begin{aligned}&\text{weight of pyrolytic noncondensable gas}\\&\quad= \text{weight of torrefied biomass} - \text{weight of pyrolytic oil}\\&\quad- \text{weight of biochar}\end{aligned} \tag{2}$$

A central composite experimental design (CCD) was employed to obtain optimum torrefaction conditions. Two factors, reaction temperature (X_1) and residence time (X_2) were selected as independent variables. The reaction temperature and time were set from 239 to 310 °C, and 16 to 44 min respectively. A 2^2-factorial CCD, with 4 axial points (factor = 1.68) and 3 replications at the center points leading to a total number of 11 experiments was employed for the optimization of the conditions of microwave torrefaction process. Table 1 shows the details of the experimental design. For statistical calculations, the variable X_i was coded as x_i according to Eq. (3):

$$x_i = \frac{X_i - X_0}{\Delta X} \tag{3}$$

where x_i is a coded value of an independent variable, X_i is a real value of an independent variable, X_0 is a central value of the independent variable, and ΔX is the step change. The second degree polynomial (Eq. (4)) was calculated to estimate the response of the dependent variables.

$$Y_i = b_0 + b_1X_1 + b_2X_2 + b_{11}X_{12} + b_{21}X_2X_1 + b_{22}X_{22} \tag{4}$$

where Y_i is predicted responses, X_1 and X_2 are independent variables, b_0, b_1, b_2, b_{11}, b_{22} and $b_{21}$are the regression coefficients.

2.3. GC/MS analysis for torrefaction oil and pyrolytic oil

The chemical composition of torrefaction bio-oil was determined by VARIANCE 3200 GC/MS system. The GC was programmed at 45 °C for 3 min and the temperature was increased at 10 °C/min to 300 °C. The ion source temperature was 230 °C for selective mass detector. The bio-oils were injected 1 μl. The compounds and their quantities were identified by searching the Mass Spectral library.

2.4. Heating value of torrefied corn stover

The heating values of torrefied corn stover were determined by the Poultry lab at the University of Arkansas.

3. Results and discussion

3.1. The effects of torrefaction conditions on yields of torrefaction products

The experimental design matrix and yields of products by microwave torrefaction are shown in Table 1. The volatile yields ranged from 14.2 to 28.5% in which the torrefaction oil and noncondensable gas contributed 5.2–17.85% and 6.93–16.01% respectively. The torrefied biomass yields ranged from 71.5 to 85.8%. This is much higher than the torrefaction oil and non-condensable gas yields. It means that most of the mass of corn stover was maintained in the torrefied biomass.

The volatile yield, oil and noncondensable gases from torrefaction process increased with the temperature and residence time. The maximum yields were observed at relatively high reaction temperature and long reaction time. That is because more components decomposed under severe torrefaction conditions [14,27]. On the contrary, the yield of torrefied biomass decreased with the reaction temperature and time and the maximum torrefied yield of 85.85% was observed at mild torrefaction conditions.

Using the experimental results three linear model equations for volatile yield (Eq. (5)), torrefaction bio-oil yield (Eq. (6)) and torrefied biomass yield (Eq. (7)) as a function of reaction temperature (X_1, °C) and reaction time (X_2, min) were obtained

$$Y = 20.94 + 5.05 \times X_1 + 0.59 \times X_2 \tag{5}$$

$$Y = 11.32 + 3.80 \times X_1 + 1.02 \times X_2 \tag{6}$$

$$Y = 79.06 - 5.05 \times X_1 - 0.59 \times X_2 \tag{7}$$

The parameters shown in the models were all significance (P-value < 0.05). In these three models no significant interaction between reaction temperature (X_1, °C) and reaction time (X_2, min) was found. The correlation coefficients of determination (R-square)

Table 1
the details of experiment design and results of microwave torrefaction process.

Run #	Torrefaction conditions		Yield of products		
	Reaction temperature X_1 (°C)	Reaction time X_2 (min)	Torrefaction oil	Torrefied biomass	Noncondensable gas
1	250	20	5.2	85.08	9.72
2	300	20	12.92	75.3	11.78
3	250	40	7.92	83.5	8.58
4	300	40	15.66	73.1	11.24
5	239	30	7.27	85.8	6.93
6	310	30	17.85	71.5	10.65
7	275	16	10.75	80.2	9.05
8	275	44	12.66	79.54	7.8
9	275	30	12.32	77.9	9.78
10	275	30	12.05	79.8	8.15
11	275	30	9.93	77.91	12.16

for three models were 0.97, 0.92 and 0.97 respectively. This indicates that these models can be used to predict the product yields for biomass microwave torrefaction process (Fig. 1).

3.2. Heating value of torrefied corn stover

The heating value of raw corn stover was 16.8 MJ/kg. Compared with the raw corn stover, the heating values of torrefied corn stover were improved to 18.64–22.22 MJ/kg depending on the process conditions. The heating values of torrefied biomass were significantly greater than those of raw biomass and similar to those of coals. Based on the mass yield and heating value of torrrefied biomass, the energy yields of torrefied biomass from different torrefaction conditions were calculated. The energy yields of torrefied biomass ranged from 87.03 to 97.87%, varying with the reaction temperature and time. The energy yields decreased with the increase of reaction temperatures. The high energy yields for torrefied biomass were obtained at relatively low reaction temperature, which indicate that a mild torrefaction temperature favors energy retention in torrefied biomass.

3.3. The effects of torrefaction on the products distribution of pyrolysis

A total of 11 samples of torrefied biomass were further pyrolyzed in the microwave reactor. The pyrolytic oil yields ranged from 15.59 to 26.67%. The noncondensable gas yields ranged from 36.94 to 45.92% and the biochar yields ranged from 32.18 to 42.03%. Compared with raw corn stover pyrolysis, the pyrolytic oil yields from torrefied biomass decreased while the noncondensable gas and biochar yields increased.

Figs. 2 and 3 show the respective effects of reaction temperature and time of torrefaction on the product distribution of pyrolysis. It was found that both reaction temperature and time of torrefaction have great effects on product distribution of pyrolysis. The noncondensable gas and biochar yield slightly increased with the increase of torrefaction severity. The pyrolytic oil yields decreased with the increase in torrefaction temperature and time, similar to the observation in previous reports [20,16,17,28,29]. The relatively low pyrolytic oil yields are due to the decomposition of components especially for hemicellose in torrefaction. If the torrefaction oil was counted, the total bio-oil yields obtained from this integrating process of torrefaction and pyrolysis were from 24.1 to 31.6%, which are very close to those from raw biomass pyrolysis [24]. The optimum conditions for total bio-oil production were at the torrefaction temperature and time of 275–300 °C and 20–30 min.

3.4. GC/MS analysis for torrefaction oil and pyrolytic oil

Both condensable volatiles from torrefaction and pyrolysis were collected separately and analyzed by GC/MS in order to understand the effect of torrefaction conditions on biomass decomposition and investigating their potential utilization. The chemical compositions of torrefaction and pyrolytic oil from different process conditions are shown in Figs. 4 and 5.

The torrefaction oils consisted mainly of ketones/aldehydes, furan derivatives, organic acids, and phenolics. Ketones/aldehydes and furans constituted a large amount chemicals in torrefaction oil which contributed about 25.4–62.4 area% and 15–35.9 area%. Organic acids contents in the torrefaction oils were about 1.5–3.5 area%. They were made up of hexanoic acid, acetic acid and propanoic acid. In torrefaction oil phenolic compounds were also observed in the range from 9.7 to 26.3 area% and increased with the increase of reaction temperature and time. It indicates that lignin also partially decomposed in torrefaction process especially at severe torrefaction. These results suggest that torrefaction oil contains high value chemicals and has potential application as fuels or chemicals.

To understand the effects of the torrefaction process on biomass pyrolysis, the pyrolytic oil from torrefied corn stover pyrolysis were also characterized. The main compositions detected in pyrolytic oil are hydrocarbons, phenols and guaiacols, and ketones/aldehydes. Srinivasan et al. [30] and Ren et al. [16,17] reported that the aromatic hydrocarbons yields were improved in torrefied woody biomass pyrolysis. In this study it was found that the hydrocarbons range from 9.1 to 26.7 area% and increase with the increase of torrefaction temperature and time. A much higher number of hydrocarbons were observed on the torrefaction condition of 300 °C and 40 min than that observed in raw biomass pyrolysis. The phenols and guaiacols range from 14.4 to 34.9 area% and 7.3–24.1 area% respectively. Relatively high concentrations of phenols were observed at torrefaction temperatures between 250 and 300 °C and time between 20 and 30 min, but relatively low concentration of guaiacols were found at these conditions. These results imply that torrefied biomass pyrolysis tends to form phenols and hydrocarbons. The relatively low yields of phenols observed at very high torrefaction temperature and long torrefaction time might be due to lignin decomposition and carbonization in torrefaction process. Therefore, the relatively low torrefaction temperature and short torrefaction time will help maintain lignin and improve the phenols production in torrefied biomass pyrolysis.

In this study, about 11.7–34 area% ketones/aldehydes were also observed. The furans observed in pyrolytic oil from torrefeid biomass pyrolysis show a decreasing trend with the increase of torrefaction temperature and time. This might be due to the lower hemicellulose content in torrefied biomass [20,28]. Compared with

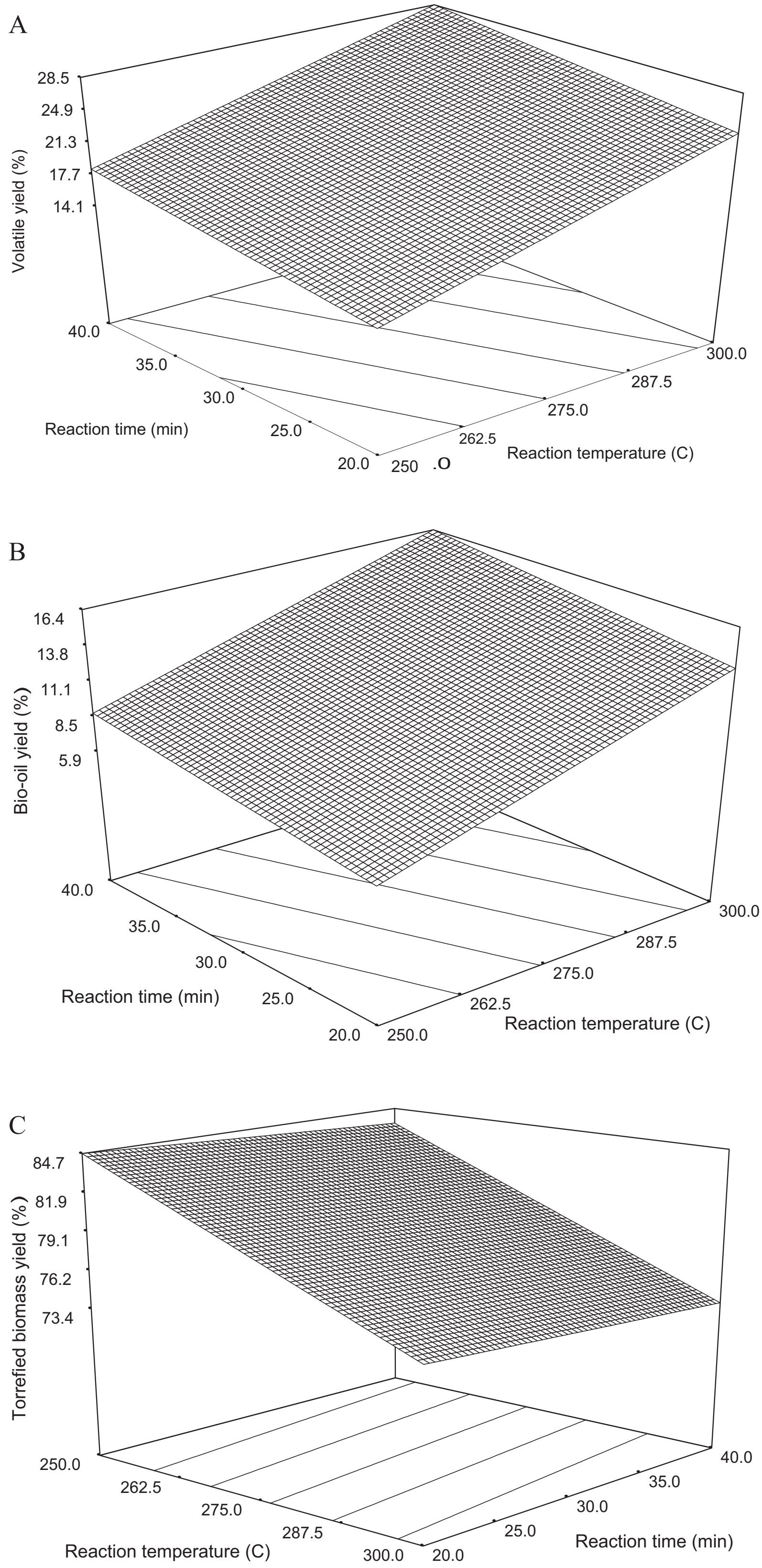

Fig. 1. The response surface profiles of volatile yields (A), bio-oil yields (B), and torrefied biomass yields (C) as a function of reaction time and reaction temperature.

raw biomass pyrolysis, torrefied biomass pyrolysis produced less organic acids which are generally lower than 1.2 area%. The pH values of pyrolytic oil from torrefied biomass pyrolysis range from 3.06 to 3.5 which are higher than the bio-oil from raw biomass pyrolysis. The pH values also increase with the increase the severity of torrefaction. These results are consistent with previous reports [28,29]. Pyrolytic oil from torrefied biomass is less corrosive which may make handling and storage easy.

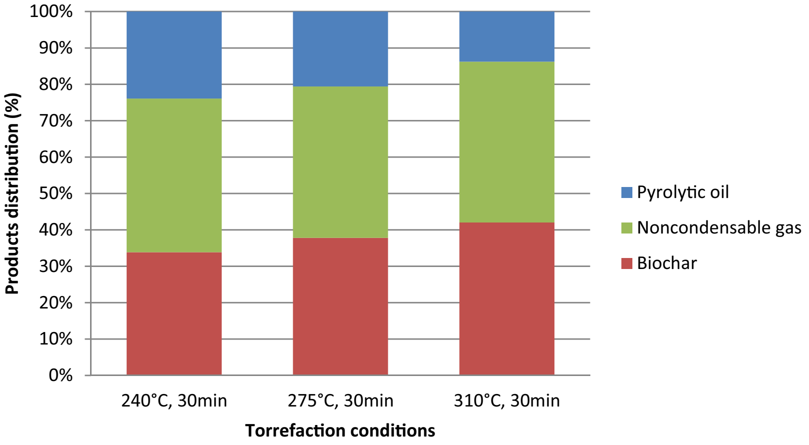

Fig. 2. The effects of torrefaction temperature on pyrolysis products yields.

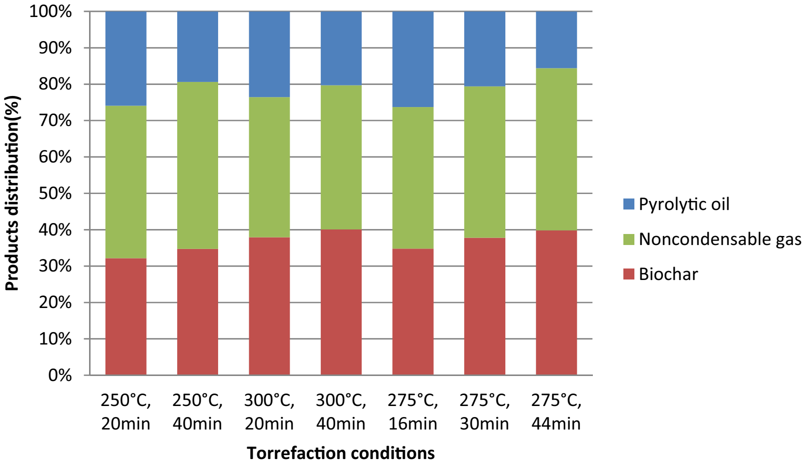

Fig. 3. The effects of torrefaction time on pyrolysis products yields.

3.5. *Mechanism analysis of torrefied corn stover pyrolysis*

The torrefaction process changes the biomass in both physically and chemically. Torrefied biomass contains less hemicellose compared to the raw biomass. The cellulose and lignin in torrefied biomass are also modified and activated during the torrefaction process [13,28]. Ibrahim et al. [31] reported that the R—OH groups decrease and C=O groups increase in torrefied biomass. These changes make the pyrolysis behavior of torrefied biomass different to the raw biomass. Zheng et al. [30] pointed out that

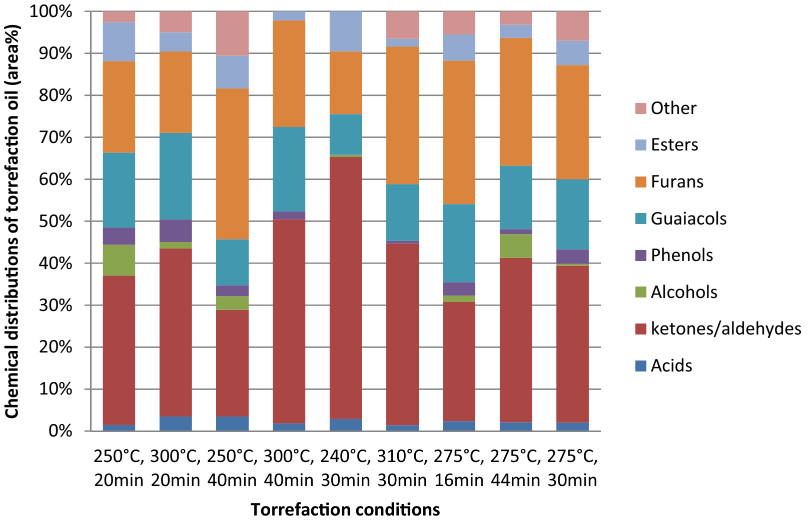

Fig. 4. Chemical compositions of torrefaction oil from GC/MS analysis.

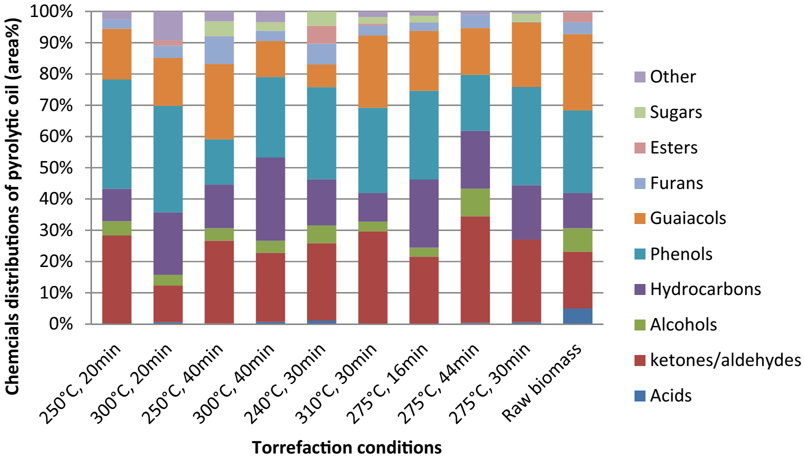

Fig. 5. Chemical compositions of pyrolytic oil from GC/MS analysis.

the devolatilization, crosslinking and charring of biomass during torrefaction has negative effects on the bio-oil yields. The same observations were found in this research that the biochar yield is increased and pyrolytic oil is decreased. However, the torrefaction process improved the hydrocarbon and phenols production in the bio-oil. It might be due to the cleavage of methyl from $O—CH_3$ in lignin decompositions and the vapor cracking by char forming in the torrefaction and pyrolysis [16,17].

4. Conclusions

This study investigated the integrated process of torrefaction and pyrolysis of corn stover. The effects of torrefaction conditions on both torrefaction products and pyrolysis products were studied. The torrefaction oils contain high value chemicals such as furans and phenolics which have potential for utilization as fuel or chemical source. The torrefaction process has negative effects on pyrolytic oil yields but improves its quality by increasing hydrocarbons, phenols and reducing organic acids. The total bio-oil yields from torrefaction and pyrolysis at the optimum torrefaction condition are close to those form biomass direct pyrolysis. The pH values of pyrolytic oils are also increased after the torrefaction process which makes the bio-oil less corrosive. Therefore, torrefaction as pretreatment can be combined with the pyrolysis to improve bio-oil quality. The next step for this research will be on reducing the carbonization of biomass in torrefaction and improving pyrolytic bio-oil yield.

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